A high temperature mechanical study on PH 13-8 Mo maraging steel

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Abstract

High temperature mechanical measurements were conducted to study the effect of the dynamic precipitation process of PH 13-8 Mo maraging steel. Yield stress, ultimate tensile strength, total elongation, hardness, strain rate sensitivity and activation volume were evaluated as a function of the temperature. The dynamic changes in the mechanical properties at different temperatures were evaluated and a balance between precipitation hardening and annealed softening is discussed. A comparison between hardness and yield stress and ultimate tensile strength over a temperature range from 300 to 600 °C is made. The behavior of the strain rate sensitivity was correlated with the intermetallic precipitates formed during the experiments.

Keywords

Mechanical characterization; nanoindentation; martensite; aging; hardening; precipitation.

1. Introduction

Maraging steels have been used in the past for a variety of different applications such as aerospace applications, tools, bearings, medical and dental equipment [1-3]. High temperature precipitation hardening of steels leads to the formation of different precipitate structures in the temperature range from 400 to 600 °C, which have been studied previously on an assortment of different alloys [3-6]. PH 13-8 steel has had a special interest among the many maraging steels due to the controlled precipitation of β -NiAl precipitates [4-6]. Precipitation hardening on PH 13-8 has been previously studied by atom probe tomography (APT), transmission electron microscopy (TEM), x-ray diffraction (XRD), Vickers hardness and tensile tests [4,7-11]. In addition, different surface treatments have also been studied on this steel such as plasma nitriding [3,12] and carburizing [12], and recently under ion irradiation [13]. However, the mechanical properties at high temperature have not been investigated yet.

Nanoindentation at high temperature (HT) has been used for the last ten years and it has become a powerful technique to measure the mechanical properties at high temperatures such as hardness (H) and reduced modulus on a variety of different materials up to 700 and 750 °C [14,15], but also other mechanical properties such as strain rate sensitivity (SRS or m -value) and activation volume (V^*) [16-19]. In recent studies, the latter properties have been used to study the governing mechanism of deformation by different nanoindentation methods, such as strain rate jump, constant strain rate or constant load [16-18,20].

This investigation focuses on the mechanical properties at high temperature of PH 13-8 steel by nanoindentation and tensile tests. Using nanoindentation, the hardness, strain rate sensitivity and the activation volume have been measured up to 625 °C. Moreover, the yield and ultimate tensile strength and tensile elongation have been studied by high temperature tensile tests up to 800 °C. The aim of conducting both kinds of mechanical tests at high temperature (tensile testing and nanoindentation) is to find a correlation between the testing techniques and the precipitation hardening process.

2. Experimental procedure

2.1. Material and microstructural characterization

The chemical composition of PH 13-8 Mo maraging steel, also known under the brand name “Corrax” from Bohler-Uddeholm Corp, Austria is shown in Table I [21]. The as-received material was in the solution-annealed state, after a heat treatment at 850 °C for 30 min and subsequent air-cooling to room temperature (RT). This resulted in a hardness of 34 HRC. The material was sliced into 0.4’’ × 0.24’’ × 0.08’’ plates and then polished with the last step of 0.1 µm diamond. The chemical products used for the etching solutions were: HCl (122 ml), HNO₃ (6 ml), Ethanol (122 ml), FeCl₃ (8.5 g) and CuCl₂ (2.4 g), for 240 s. XRD measurement was made using Cu Kα (1.54 Angstrom) radiation in a Bruker Advance D8 diffractometer working at 40 mA and 40 kV and with a step size of 0.02 °θ.

2.2. High temperature nanoindentation procedure

The prepared specimens were mounted on an indentation hot stage with high temperature cement (Omega-bond 700, Omega Engineering Inc., Stamford, USA) to subsequently perform nanoindentation and indentation creep testing at different temperatures using a Micro Materials Nanotest Platform 3 (MicroMaterials Inc, Wrexham, UK) instrument. The nanoindenter used for this study is located inside an environmental chamber, which allows purging with different gases to establish a protective atmosphere during the experiments. The chamber was purged and backfilled first with Ar and subsequently with an Ar ± 5% H₂ mixture gas. The oxygen content inside the chamber was monitored using a QRAE II gas sensor. The oxygen levels were kept between 0 and 1% while indenting. During the actual nanoindentation test, the gas purging was temporarily suspended to avoid any noise or thermal drift disturbance in the measurements. The indentation measurements were performed with a dwell time of 5 seconds while indentation creep was conducted with a dwell time of 200 seconds at the maximum load. For each target temperature the heating rate was set to 1.6 °C/min with a thermalization period of 1 hour per each 100 °C above RT, following the procedure described by Everitt *et al.* to get an isothermal contact between the indenter and the surface [22]. The distance between tip and sample during heating was set to 20 µm since this provides enough distance to account for expected thermal expansion during heating as well as it is close enough to allow for temperature equalization between tip and sample. After each indent in the maraging steel, a thermal drift assessment was

carried out. For this procedure the tip was unloaded to 10% of the maximum load applied and held for 60 seconds while the displacement was recorded. This procedure allowed us to observe that the average drift rate did not exceed 0.20 nm/s.

For this study, a cubic boron nitride (c-BN) Berkovich indenter was utilized due to its chemical stability at temperatures above 400 °C [23]. Nanoindentation measurements at different depths on fused silica were carried out prior to the experiments on the maraging steel in order to obtain an appropriate area function, which describes the tip geometry. The hardness as a function of temperature obtained from the nanoindentation experiments was calculated following the Oliver & Pharr method [24]. The distance between individual indents was greater than 25 μm to avoid any influence from neighboring indents. The penetration depth resulting from the constant load of 25 mN varied between 300 – 500 nm depending on the temperature. These rather large indents are assumed to sample a representative volume of all present phases in the material (martensite, precipitates and reverted austenite). Three temperature cycles labeled as T1, T2 and T3 (see Table II) were carried out. For the cycles T1 and T3 as-received samples were used, while for cycle T2 the sample after T1 cycle was tested. The detailed heating procedures are shown in Fig. 1a. Details about the test parameters are listed in Table II.

2.3. High temperature tensile tests

High temperature tensile samples were prepared, utilizing Electric Discharge Machining. The sample geometry is shown in Fig. 1b. The hot tensile tests were performed using an Instron machine with an integrated furnace in high purity air. Tensile tests were conducted from 100 °C to 700 °C at strain rates of 10^{-3} /s. The samples were held at the testing temperature for 30 min before beginning the tensile test and were then taken to fracture. Additionally, 2 pieces of samples were held at 500 and 550 °C for 4 hours before the test. To determine the yield stress, a 0.2% offset was used and the ultimate tensile strength was taken from the maximum stress on the flow curve.

3. Results

3.1. Microstructural characterization of the original sample.

The original as-received steel has been first studied by optical microscopy and XRD. An OM image of the original steel is shown in Fig. 2a. Similar microstructure has been shown by Bruhl *et al.* [12]. The XRD diffractogram from the original steel is plotted in Fig. 2b. The peak with the highest intensity at 44.5 is attributed to the diffraction of (011) of the bcc phase. The main peaks identified are associated with the reflection of the bcc phase (011), (002) and (112). Similar diffractograms could be seen in untreated Corrax in [3,12]. A small diffraction associated with the fcc phase (111) could be found. The lattice constant of the bcc lattice has been calculated and it is 0.2871 Å. The intensity of the bcc (011) and the fcc (111) reflections were used to estimate the fraction of retained austenite in the alloy. Less than 2% of retained austenite is found in the as-received sample, in good agreement with the finding by Höring *et al.* and Schnitzer *et al.* before aging [4,25].

3.2. Nanoindentation hardness study at elevated temperatures

A description of the creep nanohardness test conditions was shown in Section 2.2. Fig. 3a shows a representative load-time curve for a test carried out at 525 °C. As it can be seen, a load of 15 mN was applied for 200 seconds in order to study the creep of the samples at high temperature. Fig. 3b shows representative load-displacement curves obtained at different temperatures during T1 cycle. The curves obtained at 25 and 300 °C are very similar, however, a big difference could be seen starting at 400 °C. Observing the part of the graph with load constant we can see easily the significant differences of the creeping behavior at different temperatures. It looks like the curves do not show an extensive creep at RT or 300 °C. However, the creep tends to increase with the temperature over 400 °C. The differences in the creep will be further explained in the text in Section 3.3.

The hardness values obtained from the nanoindentation measurements during the temperature cycles T1, T2 and T3 over temperature are presented in Table II and plotted in Fig. 4a, 4b and 4c, respectively. Moreover, the hardness of the samples at RT after the HT nanoindentation are plotted with open symbols. For the cycle T1 (Fig. 4a), an increase in hardness can be observed from 4.2 GPa at RT, to 4.6 GPa at 300 °C and 4.4 GPa at 400 °C. After 400 °C the hardness decreased rapidly to 3.3 and 3.0 GPa at 500 and 525 °C respectively. Fig. 4b

displays the hardness of the same specimen during the heat treatment cycle T2 starting from RT. In this case no initial plateau was observed up to 400 °C but the hardness decreases constantly with temperature. The hardness measured after the 600 °C heat treatment is found to be lower than the previously measured (4.8 GPa). The temperature cycle T3 also starts with the solution-annealed sample, like T1, and the data is shown in Fig. 4c. A slight increase in hardness was observed between the temperatures of 300 to 450 °C, followed by a decrease at the 550 and 625 °C, which is in good agreement with T1 heat treatment. At the end of heating cycle T3 the material shows a RT hardness of 4.6 GPa. In Fig. 4d, the hardness of the as-received sample and the values of the hardness after each thermal treatment are plotted together with their maximum reached temperature for each cycle. The Vickers hardness (HV) has been calculated following the ratio found by Fischer-Cripps [26,27] in Eq. 1:

$$HV = 0.0945 \cdot H_{Berk} \quad (\text{Eq. 1})$$

As can be seen, there is an increase of the hardness after each thermal treatment, when comparing with the as-received value. The maximum reachable hardness can be found after the thermal cycle T1, which is in good agreement with the literature where the temperature for reaching the maximum hardness is about 525 °C [5,21]. Höring *et al.* [7] found HV values of 365 and 578 Kg/mm² for the as-received sample and after 3 hours thermal treatment up to 525 °C, in good agreement with the values obtained for the as-received sample and after the temperature cycle T1 (378 and 590 Kg/mm², respectively). Hardness measurements and uniaxial tensile tests carried out by Seetharaman *et al.* indicated that peak hardening occurred at about 525 °C due to the apparition of uniformly distributed spherical NiAl precipitates [9]. After cooling the sample to room temperature, the samples exposed to T2 and T3 heating profile show similar hardness values. This can be explained by the fact that both tests were carried out to similar temperatures (600 and 625 °C, respectively). However, it is noticeable the decrease of the hardness after T2, because the original H value was relatively 50 % higher than the original hardness value of the as-received sample for T3. Seetharaman *et al.* also found that tempering at temperatures higher than 525 °C, results in the formation of significant amounts of reverted austenite [9]. However, it should be noted that due to the different size scales of particular features, i.e. precipitates in several nanometers and reverted austenite in hundred nanometers (Schnitzer *et al* [4]) at most, the nanoindents placed in the material have a lateral dimension of in micrometer scale which leads to the fact that with an indent all present phases are sampled.

3.3. High temperature nanoindentation creep

High temperature indentation creep tests were conducted following the same experimental procedure as reported in [17,18,20]. The average strain of 6-10 measurements *vs.* dwell time is plotted in Fig. 5a, 5b and 5c and in Table II. Attending Fig. 5a, it can be seen that in general there is an increase of the strain with the dwell time and temperature. It is noticeable that at 300 °C there is almost no change with the dwell time. The same behavior is observed in T2 and T3 curves in Fig. 5b and 5c respectively. In this former test T3, the test carried out at 350 °C also does not shown a big increase with the dwell time. It seems like up to 400 °C the sample experiences little creep. Similar curves were shown by Huang *et al.* in an ODS material PM2000, where little strain was found up to 120 s dwell time at temperatures below 500 °C [19]. The final strain at the end of the dwell time has been plotted in Fig. 5d. T1 and T2 values has been plotted with the same color because both tests were carried out in the as-received sample, just at different temperatures. As seen in the creep curves, there is an increase of the creep with the temperature and the strain is below 0.2 up to 400 °C. After 450 °C a big increase in strain is found for T1, T3, and T2. It is interesting to note that the strain measured at in T2 is slightly lower than T1+T3 around 600 °C.

In order to better quantify the data and allow data processing an empirical equation (Eq. 2) is introduced and used to fit displacement-time curves of the creep test so the data can be evaluated further

$$\Delta h = A \cdot (t - B)^C + D \cdot t \quad (\text{Eq. 2})$$

Hence the strain rate ε' can be expressed with Eq. 3 which has been applied in [19,28,29]

$$\varepsilon' = \frac{\Delta h'}{h_0} = A \cdot C \cdot (t - B)^{C-1} + D \quad (\text{Eq. 3})$$

Where A , B , C , D are fitting parameters and t is time. Simultaneously, the displacement regime, namely as the steady-state creep stage, was used for further calculation and discussion. The SRS value m was calculated from the data obtained during the experiments using Eq. 4.

$$m = \frac{\partial \ln H}{\partial \ln \varepsilon'} \quad (\text{Eq. 4})$$

In addition, the activation volume V^* , that describes the volume that collectively takes part in the underlying deformation process, was evaluated by Eq. 5, as previously used in [16-19].

$$V^* = 3\sqrt{3}kT \left(\frac{\partial \ln \varepsilon'}{\partial H} \right) = \frac{3\sqrt{3}kT}{mH} \quad (\text{Eq. 5})$$

In Eq. 4 and 5, H is the hardness at elevated temperature, k is the Boltzmann constant and T is the temperature. The activation volume V^* is expressed in units of b^3 where b is the Burger's vector. The lattice constant for a similar material was reported by Höring *et al.* as 0.2874 nm [7], therefore b can be derived as 0.2485 nm for the (110) and [111] slip system. Table II shows the m and V^* values found for the different temperatures. Fig. 6a and 6b show the m value and V^* as a function of the temperature for T1+T3 and T2. Attending Fig. 6a, an increasing of m is observed with the temperature for T1+T3 and for T2. The major change is observed around 400 °C, where a big jump on the m is clearly seen. It appears that m reaches a maximum value around 0.350 at 550 °C, and after that point, a small decrease is observed. Again, we observed a small difference between the values from T1+T3 and T2 at 600 °C, as seen in the strain at the end of the creep (Fig. 5d). A similar decrease has been observed by Primorac *et al.* in CuNb alloys after testing at 400 °C [18].

The activation volume, shown in Fig. 6b, did not change with the temperature up to 500 °C. After that temperature, a continuous increase is found up to values of around 22 b^3 for the tests at 625 °C. It is believed, that at temperatures above 550 °C, the sample has been over-aged. Two supposition could explain such a notable increment in V^* : the precipitates that were formed below the 525 °C coarsen and a possible reverse austenite phase apparition.

3.4. High temperature tensile tests

The yield stress, ultimate tensile strength and total elongation (TE) obtained from the tensile tests over temperatures from 100 to 700 °C are presented in Fig. 7a, 7b and 7c respectively, and in Table III. The YS of this material at 100 °C is 751 MPa, and it is slightly lower than the reported value of 817 MPa at RT [8]. An apparent decrease of YS at 200 °C is shown. Results at 300 and 400 °C show a small increase with temperature. The maximum YS is found at 500 °C (758.8 MPa), after that point, a decrease to 637.5 MPa at 550 °C, 312.2 MPa at 600 °C and 161.5 MPa at 700 °C are found. A 4 hour-hold time (240 min) on the specimen brings the maximum YS to 780.5 MPa at 525 °C and a close result of 777 MPa at 500 °C. However, 4 hour-hold at 550 °C leads to a 527.7 MPa which represents a YS loss of 150 MPa. The YS start decreasing rapidly after the maximum reached at 500-525 °C.

As for the UTS in Fig. 7b, a plateau can be observed up to 300 °C for the 30 minute-hold specimens. In this case maximum UTS of 1043.6 MPa is achieved at 400 °C. The UTS above

400 °C shows a significant decrease with increasing temperature. It has to be noted that UTS of the 4 hour-hold specimen at 500 °C shows a 70 MPa drop comparing with 30 minute-hold one, and a similar loss of 62 MPa was observed for the 4 hour-hold specimens at 550 °C. This decrease of the UTS at 500 and 550 °C is slightly different from the decrease in the YS, because at 500 °C the YS did not change with the hold time, but at 550 °C, the measured YS showed a significant decrease. It looks like the YS is more affected by the holding time when the tensile tests are carried in the 500-600 °C temperature range.

If we analyze the TE in Fig. 7c, we do not observe significant differences from 100 °C to 400 °C (between 4.1 and 10%). At 500 °C the TE starts increasing up to 45.2% at 600 °C. At 700 °C, a decrease is observed up to 33.8%. No noteworthy differences are found between the samples tested with 30 minute or 4 hour-hold time.

4. Discussion

4.1. Mechanical property evolution at elevated temperatures

Maraging steels derive their strength from precipitated intermetallic phases, known as β -NiAl, while these steels can also contain small amounts of austenite. This austenite fraction could result from an uncompleted transformation of austenite into martensite during quenching from solution annealing temperature (retained austenite) or from partial reversion of martensite into austenite during aging (reverted austenite) [8]. Both processes (precipitation and austenite formation) are thermally activated and therefore understanding the effect of thermal activation on the mechanical properties is of value in order to understand the materials performance in a non-room temperature environment. The selected test material reveals a fully developed precipitation structure and a peak hardness after two hours of aging at 525 °C [21]. Schnitzer *et al.* [4] reported a loss of strength due to the high volume fraction (> 8%) of reverted austenite caused by over-aging in similar materials. However, in Ref [7] it was found that at temperatures below 600 °C only 1-2% reverted austenite can be seen due to the short aging time. Thus, it is reasonable to indicate that property changes occur mostly due to precipitation formation and matrix softening.

In this research, the hardness from the solution-annealed state does not change until a temperature of 450 °C, UTS shows a decrease at 500 °C, but the YS remains stable up to 525 °C. Interestingly the YS is reduced at 200 °C but increases gain up to temperatures of 525 °C. The

reason for this slight decrease in YS is not known but we can speculate that some of the quench induced microstructural features anneal at these temperatures while some early stage precipitate nucleus may form at temperatures up to 525 °C being responsible for the strength increase. At temperatures above 525 °C, a noticeable decrease in high temperature mechanical properties is observed. It is noted that this material does not decrease its YS or UTS up to 525 °C being more stable than other martensitic steels [30,31], or even ODS materials [19], which do experience continuous softening in that temperature range. It appears that the matrix strength follows from a careful balance between matrix softening and precipitate strengthening, because in this material no softening and even a slight strengthening is observed up to 450 °C. The precipitate strengthening has been previously studied from 450 to 600 °C by APT, and recently, it has been demonstrated at RT that under ion beam irradiation precipitate precursors can be formed [13]. No studies have shown the formation of β -NiAl precipitates below the 450 °C. Although no APT analysis is carried out here, it is reasonable to consider that even at temperatures below the peak hardening temperature, precipitation precursors not easily detectable by APT or TEM might form contributing to the high temperature mechanical property plateau and compensating the decrease in strength caused by matrix softening. Consequently, the balance between the precipitation hardening and the matrix softening at HT is quite stable up to 400 °C. This consideration is supported by the hardness vs. temperature test T2 where the material is fully hardened and continuously loses hardness over temperature without experiencing this plateau as it is observed in the solution-annealed case. At temperatures above 400 °C, a decrease in the properties is observed despite the fact that the precipitates have developed. Therefore, above the 400 °C it can be stated that the materials softening is more pronounced than the precipitate hardening effect.

The H values obtained at high temperature nanoindentation have been compared with other steels measured by similar methods. Reichardt *et al.* [32] showed a decrease on the hardness from 2.8 to 1.8 GPa in an 304 SS, and Huang *et al.* [19] a decrease from 3.82 to 3.80 GPa at 300 °C, 1.41 GPa at 500 °C and 1.43 GPa at 600 °C, in PM2000 alloy. It is worth mentioning that the hardness of the PH 13-8 Mo in this study is twice that of the PM2000 alloy at 525 °C. PM2000 is an early variant of ODS steels. The mechanical strength of ODS alloys is determined to a large amount by the oxide particle structure [19]. The oxide nanoclusters in the ODS alloy and the intermetallic nanoclusters formed here are hardly comparable but they provide a qualitative description of the role of the intermetallic nanoclusters in increasing high temperature mechanical properties in our alloy. At very high temperature, the precipitates (i.e. oxides in the

ODS steel and intermetallic in the PH 13-8 Mo) are less effective since the hardness degrades from 300 °C to 600 °C by 3 GPa in both cases.

Fig. 8a plots the values of YS, UTS and converted hardness measured with a Berkovich shaped indenter to a Vickers shaped indenter (T1+T3) marked as H vs. temperature in the same graph. It is obvious that the data shows the same trend and that the material starts losing its strength at 400 °C. However, the question of the relationship between tensile testing and hardness testing remains. In the literature a number of empirical equations are proposed [27, 33-37]. To understand where these relationships are valid it is important to mention that in a number of these comparisons irradiated materials are utilized which show only very little to no work hardening [38]. While these empirical equations are based on direct comparisons only few physics based approaches exist. Most importantly to mention the work from Tabor [39] where the tensile flow stress is compared to the hardness numbers. While Tabor's work is clearly a fundamental based approach the work here considers only qualitative relationships but are discussed in the context of Tabors rules below. Fig. 8b shows a plot of the YS and UTS vs. calculated Vickers hardness using Eq. 1 derived from nanohardness data. The direct comparison between YS/UTS and hardness measurements over temperature is the first directly comparison conducted to the best of our knowledge and provides insight into the hardness vs. YS and UTS relationship.

Observing the plotted values on Fig. 8b, it is clear that there is a linear relationship between the UTS and the HV in the 300-600 °C temperature range.

However that trend does not hold true in the case of the YS and HV in the same temperature range. In order to understand this discrepancy one has to consider when tabors rules are valid and why.

Tabor pointed out that the linear relationship with a factor of 3 is only valid for ideal plastic materials so materials with little to no work hardening. This can be understood by the fact that underneath an indenter significant flow occurs and the simple linear correlation between hardness and YS is only valid if the material underneath the indenter does not harden significantly during deformation but rather flows in a homogeneous fashion. It is stated that if a material shows significant work hardening then the flow stress at ~8-10% strain should be used rather than the yield stress. Unfortunately this is not possible in the samples here since the fracture occurs at already 5-7% strain with significant work hardening as it can be seen by the vastly different numbers of YS and UTS at the tests conducted below 500 °C. But at UTS no

significant work hardening can occur any further and therefore the UTS and hardness numbers show a linear relationship. In addition at tests above 500 °C little to no work hardening is observed (only very small differences between YS and UTS) and therefore the relationship appears to be better.

4.2. Indentation creep

An in-depth study of the SRS on Cr and bcc metals (such as Fe) was recently published by Wu *et al.* [40], where the measured m values varied from 0.02 to 0.01 with decreasing grain size from ~mm to ~ μm scale. Another study from Misra *et al.* exhibited m and V^* values of 0.06/0.13 and $48\text{ b}^3/16\text{ b}^3$ for coarse-grained (CG) and nano-grained (NG)/ ultrafine-grained (UFG) austenitic stainless steels respectively [41,42]. Fig. 6a displays the m values calculated from the creep data obtained for all three experiments. In that plot, low values are found at 300 °C. It can be seen in the plot how m increases at 400 °C and reaches a maximum at 525 °C. In the work by Misra *et al.*, the twinning mechanism was stated to be responsible for the increase of m values in UFG/CG comparing with CG steel since twin boundaries acted as obstacles and restrained dislocation climbing [42,43].

This study the low m values at temperatures up to 400 °C can be explained by the presence of the intermetallic precursors that hinder dislocation motion [8]. At 450 °C, an increase in m is visible, and it is associated with the bigger number of precipitates in the matrix. The m value gets even higher at 500 °C, reaching the maximum value of 0.30-0.35 in the range 500-550 °C. Leitner *et al.* suggested that in the early stages of aging, strengthening is associated with the stress required for dislocations to cut through coherent zones/precipitates and as the precipitates coarsen and become semi-coherent, the stress required for dislocations to cut the precipitates increases [8]. It is believed that in this region of temperature all the $\beta\text{-NiAl}$ phase precipitates. Thus, the material will change to a bimodal microstructure. Leitner *et al.* found that aging at 525 °C for 3 hours results in the formation of two independent precipitation phases ($\beta\text{-NiAl}$ and Ti-phase rich) [5]. After 550 °C, a decrease in m value is found. This behavior can be attributed to a coarsened precipitate population, where a change from a bimodal distribution to coarsened state takes place. Schnitzer *et al.* [44] suggested that the effect of amounts of reverted austenite after longer aging times could be present as austenite is free of NiAl precipitates and thus, no pinning of dislocations due to precipitates is present in the austenite. Ping *et al.* said that small amounts of fine carbides are formed in the martensitic matrix at high annealing temperatures and at 620

°C, in addition to the NiAl precipitates and carbides, reverted austenite grains were observed [10]. In this research, it is believed that the possible apparition of reverted austenite and fine carbides in the over-aged sample decreased the m -value, but still in the over-aged samples the formed precipitates remain in the matrix. The decrease in the m -value found for T2 could be explained by the expected bigger amount of reverse austenite after the two consecutive thermal cycles. Schnitzer *et al.* [44] observed that around 40% of the loss in strength during aging could be attributed to the presence of reverted austenite. Ping *et al.* suggested that the decrease in strength at higher temperatures (above 550 °C) is attributed to the decrease in the number density of the NiAl precipitates and the reversion of austenite, and also larger carbides [10]. In this research, similar decrease in the hardness has been shown after aging at 625 and 650 °C. One can speculate in the light of the relevant literature that precipitate coarsening occurs. Since the strength loss is rather dramatic, coarsening alone might not explain the pronounced property degradation alone and therefore one may speculate that the dislocation–precipitate interaction changes and dislocation climb becomes a more active mechanism. Obviously dislocation climb and precipitate coarsening can be related since both are based on vacancy motion and diffusion based mechanism. Contrary to the strain rate sensitivity, where the increase in m is observed at 400 °C, the activation volume shows a plateau of low values ($\sim 5 \text{ b}^3$) over temperatures up to 500 °C. Between 500 and 550 °C, just a small increase is observed. After that point, the V^* increase up to $\sim 20 \text{ b}^3$. This could be an indication of a remarkable change in the microstructure – presumably due to the apparition of reverse austenite, as suggested by the strain rate sensitivity. It is interesting to note that no remarkable changes are seen in the V^* until the apparition of reverse austenite.

5. Conclusion

In summary, tensile tests, in-situ nanoindentation hardness tests and creep tests at elevated temperature were conducted in PH 13-8 Mo in this work. The hardness, strain, yield stress, ultimate yield strength, total elongation, strain rate sensitivity and activation volume were determined at high temperature. The relationships between yield stress and hardness, and between tensile strength and hardness were shown for first time at high temperature.

The use of high-temperature mechanical testing techniques allows to probe the changes in the mechanical properties as the microstructure changes at different temperatures. The change in mechanical properties as a function of temperature indicated that intermetallic precipitate

strengthening effect starts to take place at temperatures as low as 450 °C. While the H, strain, UTS, TE and m start changing at 450 °C; YS and V^* did not change up to 500-550 °C. The correlation between the hardness, YS, and UTS between 300-600 °C and between 500 and 600 °C is discussed in the light of Tabors work.

An increase of m values with temperatures was presented up to 525 °C. Higher m values at elevated temperatures implied that dislocation climb dominated the deformation process. V^* did not change up to the peak temperature for the hardness of the material (525 °C), even with the continuous precipitation of the different precipitates. After that point, V^* starts increasing and it is associated to the dislocation climb mechanism and a possible precipitate coarsening and a reduction of the precipitate density over 600 °C. Quantification of the mechanical properties at high temperature of PH 13-8 provides valuable insight into how the precipitation hardening occurs and provides useful feedback for finite element modeling of its behavior at high temperature.

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Figure Captions

Figure 1. a) Scheme of the heating procedure during the nanoindentation tests. b) A drawing of the tensile specimen with size of 0.02” thickness, 0.25” gage width, 1.25” gage length, and 4.72” total length used in the experiments.

Figure 2. a) Optical microscopy image and b) XRD diffractogram of the sample as-received.

Figure 3. a) The loading sequence for a constant-load indentation creep experiment at 525 °C. b) Representative load-displacement curves obtained during temperature cycle T1.

Figure 5. Strain versus time curves of T1 (a), T2 (b) and T3 (c), and strain at the end of the dwell time for the three temperature cycles (d).

Figure 4. Nanoindentation hardness at elevated temperature in temperature cycle T1 (a), T2 (b) and T3 (c). d) Nanoindentation and Vickers hardness of the as-received and after the different thermal treatment samples vs. maximum temperature.

Figure 6. Strain rate sensitivity (a) and activation volume (b) of T1+T3 and T2 vs. temperature.

Figure 7. Yield stress (a), ultimate tensile strength (b) and total elongation (c) of high temperature tensile tests. All datasets filled with green solid symbols were from specimens with 30 minute-hold time at temperature while orange open ones had a 4 hour-hold time.

Figure 8. (a) Yield stress, ultimate tensile strength and the converted Vickers hardness H vs. temperature. (b) Yield stress and ultimate tensile strength vs. Vickers hardness H.

Table Captions

Table I: Chemical composition of PH 13-8 Mo maraging steel.

Table II: Test parameters and mechanical properties obtained by HT nanoindentation for the temperature cycles T1, T2 and T3.

Table III. Test parameters and yield stress (YS), ultimate tensile strength (UTS) and total elongation (TE) obtained by HT tensile tests.

Figure 1a

Figure 1b

b)

Figure 2a

Figure 2b

Figure 3a

Figure 3b

Figure 4a

Figure 4b

Figure 4c

Figure 4d

Figure 5a

Figure 5b

Figure 5c

Figure 5d

Figure 6a

Figure 6b

Figure 7a

Figure 7b

Figure 7c

Figure 8a

Figure 8b

“A high temperature mechanical study on PH 13-8 Mo maraging steel.” by *Huang et al.*

Table I

Table I: Chemical composition of PH 13-8 Mo maraging steel.

	C	Al	Cr	Mo	Ni	Fe
Wt.%	0.03	1.10	12.70	2.20	8.20	Bal.
At.%	0.14	2.26	13.52	1.27	7.73	Bal.

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Table II

Table II: Test parameters and mechanical properties obtained by HT nanoindentation for the temperature cycles T1, T2 and T3.

Test parameters			Mechanical properties			
Temperature	Max. Load	Loading/ Unloading Rate	Hardness	<i>m</i>	<i>V</i> *	
(°C)	(mN)	(mN/s)	(GPa)		(b ³)	
T1	RT	20	2/2	4.17 ± 0.12	---	---
	300	20	2/2	4.65 ± 0.31	0.09 ± 0.08	1.8 ± 1.8
	400	15	1.5/1.5	4.41 ± 0.26	0.12 ± 0.06	
	500	13	1.3/1.3	3.33 ± 0.13	0.32 ± 0.05	4.4 ± 0.6
	525	15	1.5/1.5	2.98 ± 0.1	0.30 ± 0.05	5.7 ± 0.6
	RT _{after cooling}	20	2/2	6.24 ± 0.33	---	---
T2	RT	35	3.5/3.5	6.24 ± 0.33		---
	300	25	2.5/2.5	5.28 ± 0.35	0.06 ± 0.04	2.9 ± 2.9
	400	25	2.5/2.5	4.69 ± 0.69	0.12 ± 0.08	3.2 ± 2.5
	500	20	2/2	3.37 ± 0.42	0.30 ± 0.01	4.2 ± 0.5
	600	10	1/1	1.29 ± 0.39	0.26 ± 0.05	20.1 ± 6.8
	RT _{after cooling}	35	3.5/3.5	4.81 ± 0.06	---	---
T3	RT	20	2/2	3.85 ± 0.28		
	300	20	2/2	4.21 ± 0.19	0.04 ± 0.02	3.8 ± 3.6
	350	20	2/2	4.19 ± 0.37	0.03 ± 0.01	0.01 ± 0.0
	450	20	2/2	3.98 ± 0.26	0.26 ± 0.01	4.0 ± 0.8
	550	20	2/2	2.19 ± 0.09	0.35 ± 0.03	8.2 ± 1.2
	625	20	2.5/6.7	1.18 ± 0.15	0.31 ± 0.04	21.9 ± 1.2
	RT _{after cooling}	20	2/2	4.64 ± 0.37	---	---

“A high temperature mechanical study on PH 13-8 Mo maraging steel.” by *Huang et al.*

Table III

Table III. Test parameters and yield stress (YS), ultimate tensile strength (UTS) and total elongation (TE) obtained by HT tensile tests.

Tests parameters		Mechanical properties		
Temperature	Holding time	YS	UTS	TE
(°C)	(min)	(MPa)	(MPa)	(%)
100	30	751 ± 36	995 ± 13	5.7 ± 0.9
200	30	673 ± 21	992 ± 20	8.8 ± 3.4
300	30	696 ± 26	1010 ± 38	4.9 ± 0.3
400	30	746 ± 20	1043 ± 7	4.1 ± 1.0
500	30	758 ± 36	929 ± 94	14.1 ± 2.1
	240	777	858	14.1
525	240	780	816	10.2
550	30	708 ± 29	679 ± 24	26.3 ± 6.1
	240	527 ± 47	617 ± 103	31.2 ± 16.2
600	30	312 ± 16	353 ± 12	45.2 ± 23.2
700	30	161 ± 5	214 ± 5	33.8 ± 2.6